1541

TO

MY RESPECTED FATHER

THIS WORK

IS GRATEFULLY DEDICATED.

CHAPTER III.

GENERATING PLANTS FOR SULPHUROUS ACID GAS, AND SULPHITATION VESSELS FOR JUICE AND SYRUP

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Subsequently, Stolle (1838) obtained a patent upon the ground that sulphurous acid would act as a decolorizing substitute for bone black. Other investigators, including Merge, Boulon (1846) and Melsen (1849), carried out extensive experiments on the application of different sulphites, such as sulphite of lime, sulphite of alumina, etc., to this problem. The results, however, were unsatisfactory and impracticable, and consequently the idea of their application was abandoned.

The keen researches along the above lines did not remain confined to beet sugar fabrication, but soon spread to the sugar cane countries, for in the colony of Mauritius in 1865, Dr. Icery conducted successful experiments, which have been of appreciable benefit to the industry in that island. His method was shortly afterwards introduced into Java.

Opinions as to the value of sulphurous acid as a purifying agent, and the most effective method of its application, were for a long time by no means concordant. The next patent, granted to Seyferth (1869) was issued upon the basis of introducing the acid directly into the vacuum pans during the boiling of syrup. This process came into vogue in Germany and France, but certain technical difficulties caused it to be gradually abandoned.

Numerous subsequent experiments, extending over a number of years, have elucidated to a great extent the problem encountered in applying sulphitation processes. Prominent among the numerous investigators who have been engaged in this field of research are Basset, Fradiss and Schulze. It was not till 1884, however, that the sulphitation process in sugar fabrication was finally established. This was chiefly due to the exhaustive researches of Battut.

In the sugar cane countries, where this process has made rapid strides, and is at the present day playing the most important part in the manufacture of superior white plantation sugar, it is evident that it has been, ever since its establishment, the subject of extensive experiments and considerable improvements. During recent years the knowledge of this process in cane sugar fabrication has been greatly extended, notably as the result of valuable researches by Dr. Zerban, Harloff, Hazewinkel, and others.

tank. This heating process is kept up for some five hours, during "which time volatile impurities of low boiling point will have "escaped."

"Subsequently the fluid mass is discharged into a closed furnace, "or so-called 'cornue,' in which its temperature is further raised "to 400° C by means of coal. The distillation of the sulphur is "conducted by leading the sulphur vapours into a cooling chamber. "The temperature prevailing in the interior of this chamber is "gradually brought to about 130° C, so that the distillate accumu- "lates at the bottom as a liquid, which is tapped off from time to "time to be cast into the customary form of rods."

Nature and Properties.—Sulphur exists in several allotropic modifications, but the most important one in our case is the ordinary or rhombic sulphur, the properties of which are therefore dealt with. Commercial roll sulphur has an average specific weight of 1·92 to 2·0. Its composition is naturally subject to considerable variations. Italian and French sulphurs usually contain 96·3 to 99·1 per cent. of sulphur, 2·4 per cent. to 0·2 per cent. of ash and 0·1 per cent. of arsenic. An official analysis of the Javanese sulphur gives the following figures:—Sulphur 99·8 per cent., moisture 0·04 per cent., ash 0·02 per cent., arsenic absent. Obviously this kind of sulphur is exceptionally pure.

Commercial sulphur forms yellow crystals which melt at 113° C, ignite in air at 250° C, and boil at 445° C under ordinary pressure.

On ignition the sulphur burns with a characteristic blue flame; just above the boiling point the vapour is orange yellow in colour but on continued heating it darkens, becoming deep red at 500° C, while at higher temperatures it again lightens, becoming straw-yellow at 650° C.

Other interesting phenomena are witnessed when sulphur is heated above its melting point. The solid melts to a pale yellow liquid, which on continued heating gradually darkens and becomes more viscous, the maximum viscosity occurring at 180° C, the product being then dark red in colour. On further heating the viscosity diminishes, while the colour remains the same.

At the same time, small quantities of sulphur trioxide (SO_3) are formed, which render the gas obtained by this combustion more or less foggy.

Properties of Sulphurous Acid.—Before proceeding further, it may be well to consider those properties of sulphurous acid which may play an important part in the process of its generation and practical application.

Sulphur dioxide is a colourless gas, possessing a characteristic suffocating odour. It is more than twice as heavy as air, its specific gravity being 2·26. It is readily soluble in water, its solubility at various temperatures being as follows :—

1 volume of water at 0° C dissolves					79·79	vols.	SO_2
1	„	„	20°	„	39·37	„	„
1	„	„	40°	„	18·77	„	„

The solution is strongly acid, and is regarded as sulphurous acid, the gas having entered into chemical union with the water :—

$$SO_2 + H_2O = H_2SO_3$$

When sulphurous acid gas is heated to 1200° C it entirely decomposes into oxygen and sulphur, thus :—

$$SO_2 = S + O_2$$

In contact with an excess of oxygen, the sulphurous anhydride will be transformed into sulphuric anhydride :—

$$SO_2 + O = SO_3$$

These two latter actions are of considerable importance, being detrimental to the successful employment of SO_2 as a purifying agent in sugar manufacture.

According to experiments conducted by Horsin-Déon, the dissociation takes place even below 1200° C, namely, at 800°-900° C. When this temperature is reached, a series of dissociation processes occur, *viz.*, first of all, SO_2 into S and O_2, and this excess of oxygen enters into combination with SO_2 to form SO_3; and again SO_3 into SO_2 and O.

When dealing with humid gas, the unavoidable formation of

Generation of Sulphurous Acid.

Precautionary Measures to be observed.—The essential points to be borne in mind in connection with the generation of sulphurous acid gas, and the construction and arrangement of sulphurous acid producing plants, are comprised in the following.

The sulphur is almost exclusively burnt in a cast-iron combustion chamber, into which the air necessary for the combustion may be introduced either by compression or suction.

Air Regulation.—The regulation of the quantity of air is of great importance, for we have seen that lack of air involves an incomplete combustion of the sulphur, which in its turn promotes the occurrence of sublimated sulphur in the pipings, and on the other hand, an excess of air renders the saturation gas generated too dilute, which affects the sulphitation process.

Taking the latter point into consideration, therefore, it is important that the sulphur furnaces should always be kept within reasonable dimensions, otherwise an excessive volume of air would be necessary for keeping the sulphur burning; at the same time it is desirable to establish a correct relationship between the size of furnace and the air compressor.

Combustion Temperature.—Excessive combustion temperatures should be avoided; otherwise, as previously stated, dissociation of the sulphurous anhydride is apt to occur, in which case a certain amount of sulphur in the form of vapour is entrained in the pipings, and on cooling becomes re-crystallized, ultimately causing the pipings to become choked.

Sublimators. In spite of all care and supervision, this phenomenon, called sublimation, is bound to occur to a more or less extent, hence it is imperative to provide such installations with a sublimator, fitted with an effective water-cooling device.

Drying the Air.—Further, it is essential that the air should be dried prior to entering the combustion chamber.

Although it has been repeatedly proved that humid air incontrovertibly promotes the formation of sulphuric acid, and in spite of

CHAPTER III.

GENERATING PLANTS FOR SULPHUROUS ACID GAS.

Sulphitation Vessels for Juice and Syrup.

There are a great number of types of installations for the generation of sulphurous acid gas. The simplest, but at the same time most primitive installation imaginable, is such as is still to be met with in a number of factories in Mauritius.

The plant usually consists of a couple of miniature chimney-like iron furnaces, the top-ends of which are connected with the sulphitation vessels by means of piping. The air required for the combustion of the sulphur is drawn into the combustion chamber by means of an injecting arrangement (Giffard), which carries the sulphur fumes forward directly into the sulphitation vessels by means of steam.

Owing to the absence of gas purifying and cooling devices, it is evident that this manner of operation involves a generation of impure saturation gas, in addition to the occurrence of sulphuric acid and sublimation products. In a few cases gas washers and sublimators are employed.

Another apparatus met with in a few factories on that island, which appears to be appreciated owing to its simplicity, cheapness, and alleged efficiency, is shown in Fig. 1, a description[1] of which is as follows :—

The installation is composed of

A sulphitation tank A.

A cast iron furnace B, provided with a slide arranged in such a manner as to reduce the amount of air entering the chamber to a minimum.

A refrigerating sublimator C.

A washer D, which consists of a rectangular vessel of solid antimonized lead, divided vertically into two compartments by means

[1] Baissac, Société des Chimistes de Maurice, August, 1912.

of a leaden partition, leaving a communicating space between the compartments of one inch from the bottom. This arrangement enables the washing of the sulphurous gas to be carried out. The gas is drawn by an injector E through the water, thus ridding it of its impurities. Juice inlet is 1, juice outlet 2, the washed gas enters injector chamber by 3, and steam by 4.

The gas washer contains about 20 litres of water, which is changed at intervals of eight hours. The quantity of sulphuric acid contained in the water, according to analyses taken on different occasions, varied from 16 to 28·6 grms. per litre, giving an average of 20·3 grms. of SO_3, or 100 grms. per washer during eight hours' work. At the end of the milling season, a greyish powder is found to adhere to the partition, principally composed of sulphates and sulphites of lead, silicon, traces of iron, etc.

The main advantages attributed to the gas washer are that it retains the SO_3 formed during the combustion process, and also the impurities of the sulphur which have not been deposited in the sublimator.

Quarez Sulphitation Installation.

We now come to an installation which enjoys a wider range of application, and may be met with in beet sugar as well as in cane sugar factories, namely the Quarez Sulphitation Plant. It belongs to the class of continuous sulphitation apparatus, and owing to its simplicity as well as efficiency, it is much appreciated.

The installation consists of an ingenious combination of an air dryer, sulphur furnace, sublimator, juice pump and sulphitation tank, which entire arrangement takes up but a small space.

The sulphur is burnt in a rectangular combustion chamber L. The tray on which the sulphur is charged is moveable for the purpose of introducing the latter, and also for cleaning; the door may be opened without the sulphur fumes escaping into the atmosphere. The air is drawn from underneath through an air drying device S, composed of a chamber containing several layers of quicklime, and thence into the combustion chamber.

the non- or slightly sulphitated juice up and forces it into the injector F, whence it circulates downwards through the column G, drawing the sulphurous acid gas with it into the larger division of the tank. The juice is thus continuously kept in circulation, until the required degree of acidity is obtained. The actual sulphitation process therefore occurs in the vertical column, where the gas and juice are intimately mixed.

The gas which has not been absorbed will bubble through the juice in the tank. The sulphitation tank is provided with a test box P_1 and a run-off pipe R.

The sulphitation process is controlled by regulating the quantity of juice to be treated and the speed of the pump.

The points in connection with the Quarez installation put forward by its advocates are :—

1. The sulphurous acid gas is introduced into the juice by means of suction and not forced through by compression.

2. The quantity of juice in contact with the acid is small, namely, three to four hectolitres.

3. The entrance and discharge of the juice being continuous, the sulphitation process may be interrupted and recommenced instantaneously without any inconvenient consequences.

4. The Quarez apparatus is readily installed, occupying very little room and allowing of various combinations of the different parts.

5. Its manipulation is of the simplest nature.

6. The apparatus has no valve in contact with the sulphurous acid ; the injector is constructed of special metal, proof against deterioration due to the acid gas.

7. The quantity of sulphur consumed is very small, and no gas is allowed to escape into the factory.

Although the general advantages claimed for the Quarez apparatus justify its extensive application in sugar countries, it is evident that local conditions are bound to play an important part in the usefulness of this type of sulphitation installation.

In countries like Mauritius, Natal and Louisiana, where generally the sulphitation process occurs before tempering, this apparatus is

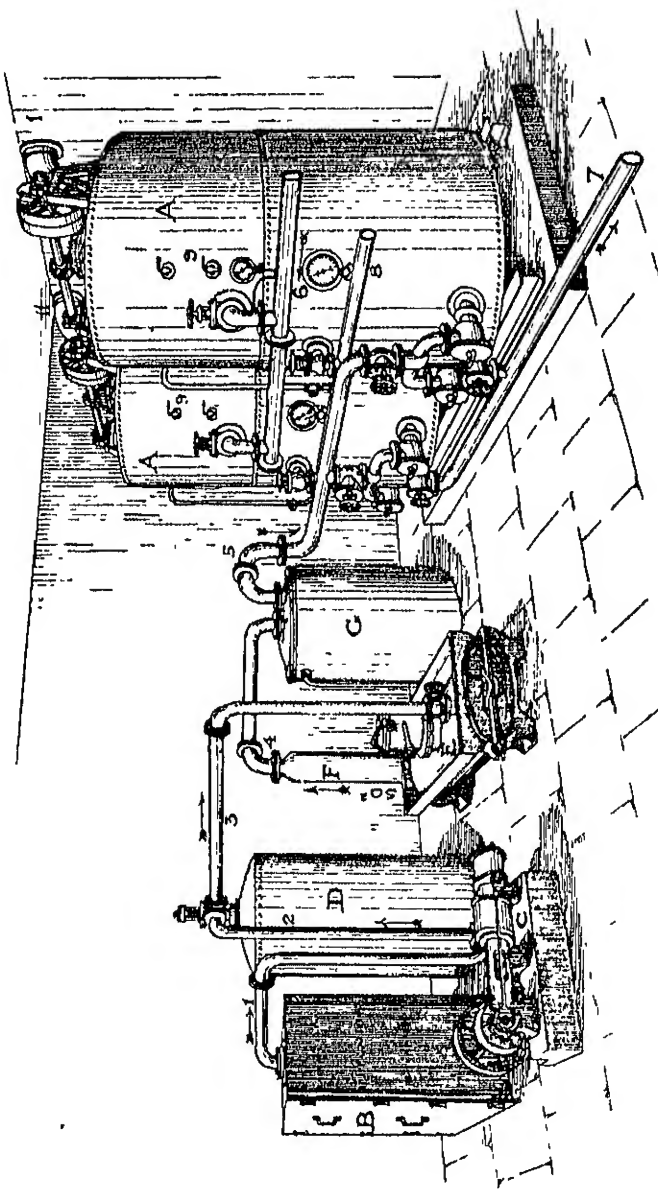


Fig. 3

Apparatus for Sulphitation as used in Java, etc.

This consists of a vertical rectangular wooden chamber **C**, of suitable dimensions, varying greatly according to the special requirements in different localities. The height of this tower ranges from 8 ft. to 12 ft. At intervals within the tower, horizontal or slanting perforated partitions or similar devices for the purpose of distributing the juice are fitted.

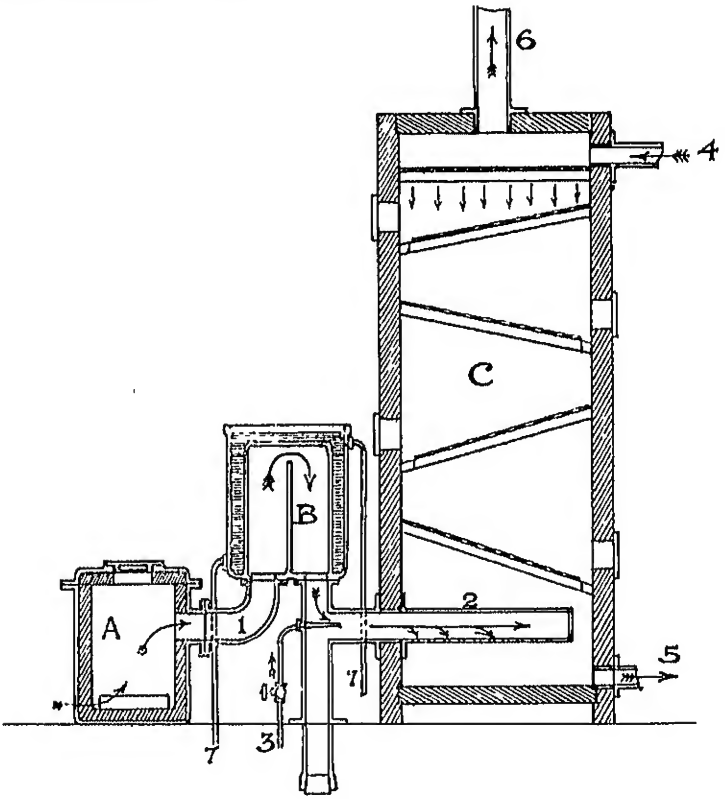
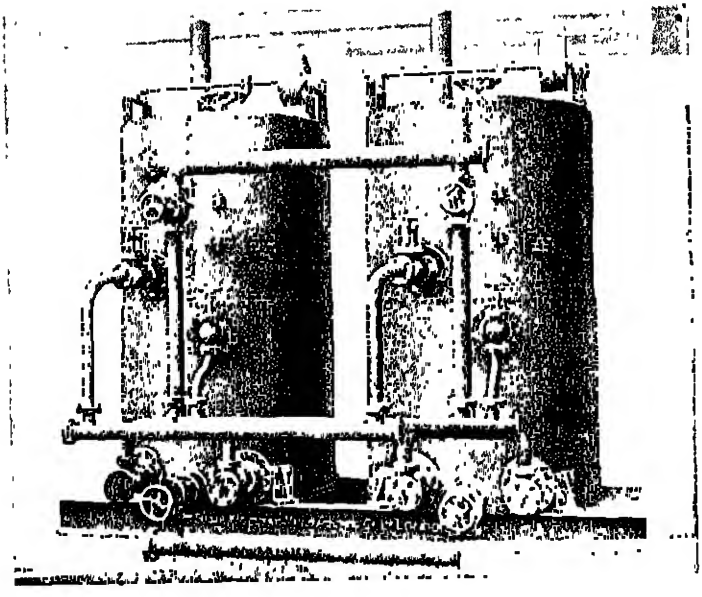


Fig 4
The "Sulphur Box"

The juice enters at the top of the tower **4** and gravitates as a fine shower, being broken up as much as possible by the partitions, whilst the sulphurous acid gas enters the box at the bottom **2**, either under suction or pressure promoted by a steam jet **3** suitably arranged. After thoroughly intermixing with the gas in its downward

Plate II



SULPHITATION VESSELS WITH STIRRING GEAR

CHAPTER IV.

THE CONTROL OF THE SULPHUROUS ACID GAS GENERATING STATION.

Quantity of Lime.—The amount of quicklime required for drying the air cannot be pre-determined, owing to the varying degrees of humidity in the atmosphere, as well as to the gradual formation of a skin of slaked lime, resulting in a decrease of drying power. For this reason, it is always advisable to place a larger quantity of lime in the air dryer than is theoretically necessary. The regular changing of the lime at certain intervals is also essential.

The Manipulation of the Sulphur Furnace is performed as follows:—Different methods may be applied to start the furnace. The requisite amount of sulphur being placed on the tray, it may be ignited by throwing upon its surface burning sulphur wicks, which are readily made by dipping ends of old ropes into melted sulphur. This method, however, often produces merely local combustions, so that when the door is closed and the compressor started, it frequently happens that the flame is soon extinguished or that the SO_2 production is poor.

Another mode of operation sometimes applied consists in first melting the sulphur rolls in the tray by burning wood under it. The advantage is that a well-distributed layer of melted sulphur is obtained, allowing of an easy ignition.

By spraying some alcohol on the sulphur, an evenly distributed ignition may also be attained.

Regulation of the Air Supply.- The next point which deserves attention is the regulation of the air supply for combustion. It is obvious that an excessive quantity of air rushing through the furnace may either be the cause of extinguishing the flames, or producing a gas of poor strength; on the other hand, an insufficient supply of air causes incomplete combustion and sublimation. By rational regulation of the speed of the air compressor, and the air valve on the furnace, a smooth working of the furnace is ensured.

sulphur on the weight of cane. This calculation is naturally based on the assumption that rational operation of the sulphitation process takes place, entailing the use of no more SO_2 than is necessary for neutralization purposes.

(2) *Juice Sulphitation before Tempering.*—In factories where sulphitation precedes the tempering of the juice, the consumption of sulphur obviously depends on the degree of acidity to which the juice is brought. Assuming this to be 0·7 grms. SO_2 per litre, and considering that according to the equation (*b*) 1 part of S produces 2 parts of SO_2, it follows that 0·35 grms. S will produce 0·7 grms. SO_2 per litre of juice. Taking the above figures of 100 parts of cane giving 90 parts of juice, we again obtain a consumption of 0·03 per cent. of sulphur on the weight of cane.

(3) *Syrup Sulphitation.*—The approximate quantity of sulphur required for the sulphitation of syrup and molasses may be arrived at in a similar way to that under (*2*).

It is evident that these figures are only approximate, as there are many factors of a complicating nature, which, if taken into consideration, would make a calculation impossible. These above methods are however, sufficiently accurate for practical purposes.

Figure 5 shows the apparatus employed for this purpose, of which the following gives a brief description.

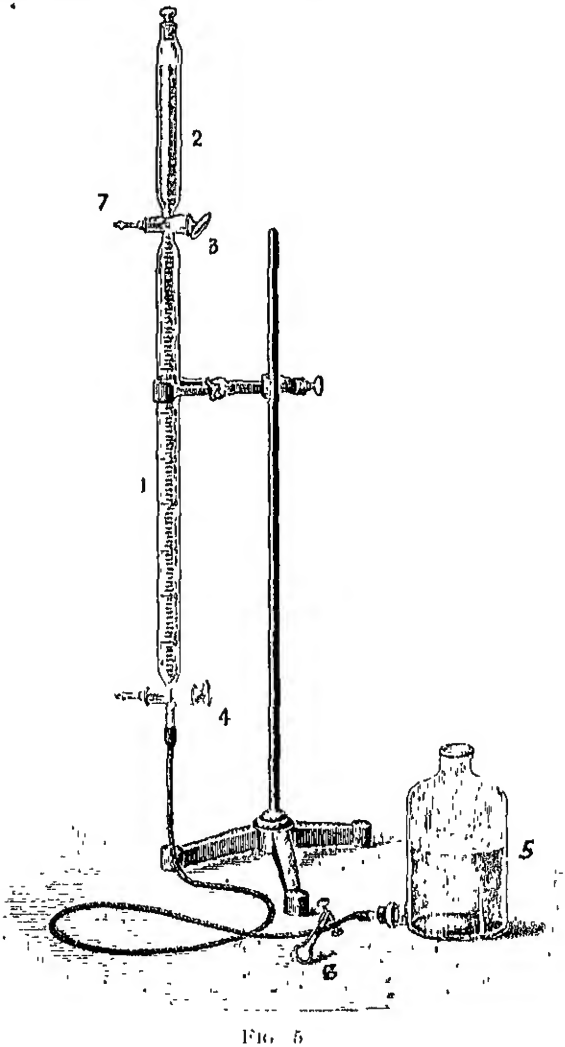


Fig. 5

Apparatus for analysing Sulphurous Acid Gas

Owing to the easy solubility of sulphurous acid gas in water, the apparatus as well as the method of application differs somewhat from that used for analysing carbonic acid.

CHAPTER VI.

ACTION OF SULPHUROUS ACID ON JUICES.

The methods of application of the sulphitation process with regard to cane juice in plantation white sugar factories are twofold, namely :—

1. After tempering the juice.
2. Before ,, ,,

The former is the standard method as applied in Java, while the latter is generally in vogue in Mauritius, and is also frequently met with in Natal and Louisiana.

Action of the Gas on Juice.—Before proceeding to discuss the *pros* and *cons* of the different methods, however, it is necessary to enter into a general study of the various properties and actions of the sulphurous acid gas in connection with the cane juice.

It is an acknowledged fact that sulphurous acid gas is a purifier, a decolorizer, a neutralizer, a reducer of viscosity, and a good antiseptic.

Purifying Action.—When sulphurous acid gas is applied to raw cane juice (mill juice), the following phenomena may be observed :- A sample of the cane juice of the typical greyish to dark green colour is treated with sulphurous acid gas in a glass cylinder to about 0·7 grms. SO_2 per litre. As soon as the sulphitation operation ceases and the liquid is again at rest, very small particles are formed in the juice, which however are soon transformed into voluminous and flocculent masses of varying density. The difference of density causes a convection, and so the heavier parts move downwards and the lighter ones upwards. After a lapse of time, varying according to circumstances, the settling process is completed. The sediment which usually occupies about one quarter of the total volume, is of a lemon-yellow to greenish-grey colour, while the juice is less opaque and of a much lighter shade.

The precipitate is mainly composed of organic matter.

oxidized by the oxygen of the air, causing the juice ultimately to assume its original colour.

By acidifying juices by means of SO_2, it may be further observed that, as long as the acidity of the juice is preserved, the decolorization is maintained. This permanent decolorization in case of acid solutions is chiefly attributed to the bleaching action of the sulphurous acid salts formed. During the different phases of evaporation processes these sulphites check, to a greater or less extent, any subsequent development of colouring and an oxidizing processes. They are easily oxidized to sulphates, in which form they are invariably found subsequently in the syrups and molasses; for example:—

$$2\ CaSO_3 + O_2 = 2\ CaSO_4$$

It is also owing to the said property that sulphites do not impart a darker colour to glucose solutions on heating.

On subsequent neutralization of the acid juices, however, the decolorization entirely disappears so that the juice assumes its original colour.

Neutralizing action.—As an agent for neutralizing the alkalinity of limed juices, the application of sulphurous acid is generally adopted. Besides being cheap, the lime-salts of this acid are practically insoluble in neutral or alkaline solutions.

Reducing viscosity.—Although the opinions of authorities with regard to the alleged property of sulphurous acid of reducing the viscosity of juices are not unanimous, in practice it is generally accepted that such an action occurs. So far, however, numerous experiments have failed to disclose such an influence.

Preservative action.—Another property ascribed to sulphurous acid is a preservative action on juices. It is generally accepted that juices and syrups are less liable to fermentation after sulphitation.

author in Mauritius, supplemented by analyses carried out at the local " Station Agronomique," gave identical results, namely, that a distinct separation of gum was observed on allowing an adequate amount of lime to act during a certain time on cold cane juice.

Pectin ingredients unite with lime to form insoluble as well as soluble compounds, which latter are also precipitated to some extent during defecation.

Colouring substances, such as anthocyan and chlorophyll, are precipitated by sufficient tempering.

The sugar further combines with the excess of lime, dissolving and forming saccharates, as long as the juice is not heated.

Besides gummy matter, the most obnoxious substances playing an important part in the manufacture of white cane sugar are the " reducing sugars," or, as they are often termed, "glucose."

Lime, like other alkaline agents, acts on the reducing sugars in the cold, forming glucosates which are analogous to saccharates. These compounds are unstable, and hence easily decomposed. At lower temperatures (below 55° C.) the chief product due to the action of lime on glucose is lactic acid, which is a comparatively stable acid and not subject to spontaneous decomposition. At higher temperatures, however, dark-coloured lime-salts of organic acids, especially glucinic and saccharic acids, are formed. These lime-salts easily decompose, forming acid substances which may cause inversion and which moreover do not crystallize, hence they retard evaporation and crystallization. Their viscosity is generally known to cause considerable trouble and losses in the boiling and curing departments.

The main actions of lime upon the various constituents of the the cane juice, therefore, may be summarized as follows

Lime neutralizes the free acids; combines with albuminoid, gummy and pectin substances, which are subsequently partly removed; precipitates various bark pigments and chromogens. On the other hand, it also forms saccharates and glucosates, depending on the temperature, the concentration both of lime and sugars, and the duration of contact.

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CHAPTER VII.

PRINCIPLES OF THE APPLICATION OF SULPHITATION TO JUICE.

Having briefly dealt with the various actions of sulphurous l on cane juice, we can now proceed to study the principles of e purifying processes by means of sulphitation.

As already stated, there exist two methods of applying the phurous acid gas, namely after and before tempering the juice.

(I) Sulphitation after Tempering the Juice.

According to this method the cane juice coming from the mills previously treated with a certain quantity of lime, a process own as "tempering" the juice. This purifying agent being no less importance than sulphurous acid in the process in estion, a study of its chemical action on cane juice is obviously lispensable.

Tempering.—The first action of lime on cane juice is to neutra- e the free organic acids, forming calcium organic salts. The mbined organic and inorganic acids may likewise be decomposed. The phosphoric acid occurring in the juice will unite to form id calcium phosphates which are precipitated as flocculent tribasic osphate.

Further, lime acts upon albuminoid, gummy and pectin matters, ich are partly precipitated. The lime combines with the albumen ich was previously suspended in the juice in a gelatinous ndition. This compound is coagulated during the subsequent eatment by the combined action of lime and heat.

With regard to the action of lime upon gummy substances, the inions of various authorities are divided. Dr. Maxwell[1] found me time ago that a large quantity of gum was separated by liming the cold. A series of experiments conducted by the present

[1]Report of the Hawaiian Exp. Station, 1896.

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BATTERY OF SULPHITATION TANKS.

It is thus evident that lime is an excellent purifying agent, and s therefore no wonder that its application as such to sugar juices been more than justified from the earliest times.

Sulphitation.—After tempering by lime in the cold, the sulphurous l saturation is commenced. By this treatment the greater part he lime compounds enter into union with the sulphurous acid to m insoluble sulphites, which, after undergoing a heating process, finally precipitated and removed with other substances in the ecators or subsiding tanks. It is evident that sulphurous acid is d in this case simply as a neutralizing agent. The clear juice m these tanks should be of a very light olive yellow colour, quite nsparent, devoid of the impurities in suspension which cause the uid to assume a turbid or less transparent appearance.

The sulphitation is continued until neutrality is reached as wn by the phenolphthalein test, described under "Indicators" at end of this chapter. If the extent of saturation were still ther carried out, over-sulphitation would occur. This would an the conversion of the insoluble sulphites, such as those of cium and potassium, into soluble bisulphites, according to the iation :—

$$CaSO_3 + SO_2 + H_2O = Ca(HSO_3)_2$$

Calcium sulphite — Calcium bisulphite

The juice would consequently be rendered acid. During heating the juice-heaters, and especially during evaporation, these soluble ulphites are split up into sulphurous acid, water and insoluble phite. The latter is deposited around the heating tubes of the iporating vessels, thus forming an incrustation which reduces nsiderably the transmission of heat. The liberated sulphurous d passes into the condensed water of the evaporators, causing rosion of the tubes.

It is therefore obvious that great care should be taken to see at the sulphitation process is not carried too far.

(II) Sulphitation before Tempering.

This method, as stated previously, is generally adopted in uritius, and is also met with in Natal and Louisiana. It is rformed in the following manner : —The cane juice from the mills is

directly treated with sulphurous acid gas until an acidity of from 0·8 to 1 per cent. of SO_2 is reached, and after this the juice is neutralized by means of lime.

While in Mauritius, the author fortunately had the opportunity of investigating the advantages and disadvantages of both methods, in the laboratory as well as in general practice, that is, in a factory which formerly worked according to the sulphitation-before-liming process, but which on the author's advice subsequently abandoned it in order to introduce the reverse method. The results[1] of these investigations, later extended to Natal practice, where both methods are in vogue, are studied in the following sections.

As we have seen, treatment of crude mill juice with sulphurous acid gas results in the production of a precipitate and a decolorization of the liquid. On subsequent neutralization of the acid juice, however, the greater part of the precipitate which is mainly composed of organic matter, is re-dissolved, and simultaneously the juice assumes its original dark colour.

We therefore conclude that the beneficial influence of the sulphurous acid on the cane juice, both from a purifying as well as bleaching point of view, disappears with the subsequent liming process, the only useful action of the SO_2 in this case being that it renders the mill juice acid for subsequent neutralization.

It would be different if the voluminous precipitate due to the sulphurous acid could be removed before the application of lime. The nature of the precipitate does not allow of a practicable filtration so that the removal involves considerable practical difficulties, but undoubtedly it has a beneficial effect upon the ultimate quality of the purified juice.

In Mauritius, one factory adopted this principle of removing the precipitate due to sulphurous acid prior to the liming, and obtained excellent results. This operation was effected in what is locally called a "bac portal," a shallow tank divided into narrow longitudinal compartments, at alternate ends of which the partitions are cut away to a depth of about 1 m., thus allowing the juice to travel

[1] The main practical results have been elaborately dealt with in an article by the author in "Bulletin de la Société des Chimistes de Maurice," *Nov.*, 1911, No. 7.

istance of about 200 feet, all the time depositing the suspended ecipitate.

Returning to the sulphitation-before-liming process, the acidified ce is subsequently limed to neutrality. Now, how does the e act under these conditions? The juice being strongly acid, tands to reason that the lime will preferably enter into union h the acid, forming insoluble sulphites, consequently the purify- property of the lime is not allowed to act sufficiently before ng rendered partly ineffective by the formation of lime salts.

Comparison.—Although, in the laboratory, scarcely any external erences can be observed in juices treated according to the two cesses, liming-sulphitation, and sulphitation-liming, this is not so he case of factory practice. In the above-mentioned mill, where h methods could be applied alternately, it has been distinctly erved that the defecated clean juice derived from the sulphitation-ore-liming process is inferior in all respects. Though the same t-olive colour may be obtained, the juice is much less transparent. rther, by regulating the quantities of sulphurous acid gas and e, this peculiar condition of the juice could not be improved.

The peculiar opaqueness of the juice (if it may be so termed) is st probably due to gummy and pectin impurities in suspension, ch have escaped the action of the lime. To enter into fuller ils regarding this subject would be exceeding the scope of the sent work, it will suffice to mention that experiments conducted both laboratory and factory all go to prove that under normal umstances the liming-sulphitation is superior to the reverse hod.

Only under abnormal conditions is the application of the sulphi-on-before-liming process justifiable, or even advisable. For ance, in Natal, in certain seasons, the glucose content of the ba" cane may rise to a proportion unheard of in other sugar e countries, so that the operation of sulphitating the juice prior empering is obviously more advantageous for the following ons.

High Glucose Proportion.—In cane juices containing a high percentage of reducing sugars, it is evident that the notorious action of lime on glucose, with the regrettable consequences pointed out previously, is readily promoted. This fact has been repeatedly confirmed by practice in Natal.

After abnormal seasons, including prolonged droughts when juices with excessively high glucose contents occur, and the tempering-before-sulphitation process is adopted, it has been frequently observed that considerable inconvenience and manufacturing losses are experienced, especially in the boiling and curing departments, the resulting strikes being usually of an inferior quality, and requiring an exceptionally long boiling time, whilst at the curing station the capacity of the centrifugals is reduced, a decrease in both output and quality of the cured sugars being observed.

Naturally under such abnormal conditions, a greater or less quantity of viscous constituents of analogous obnoxious characteristics are likely to occur in the juice concurrently with the glucose, so that one is apt to attribute the above mentioned abnormalities to these ingredients. This is obviously partly true, but nevertheless, cases frequently occur when the cane juice is comparatively devoid of such viscous ingredients, but rich in reducing sugars, the treatment of which is accompanied by the above phenomena.

By applying sulphitation before tempering, it appears that the reducing sugars are more likely to escape the detrimental action of the lime, owing to the latter entering into immediate combination with the sulphurous acid contained in the juice.

Dextran Fermentation.—Another case where the sulphitation-liming operation may be advantageously applied is the following :—

The occurrence of micro-organisms called *Leuconostoc mesenterioides* is undoubtedly familiar to those concerned with milling operations. Although in nearly all colonial sugar mills the cane juice may be to a less or greater extent infected by these micro-organisms, the extent to which Natal mills are frequently subject to them is considerable, especially after prolonged droughts.

These *Leuconostoc mesenterioides* rapidly form from sugar a atinous substance, principally consisting of "dextran," which ocess is accompanied by a strong formation of acetic and lactic ds. A small deposit of this dextran is apt to grow very rapidly a current of alkaline juice. A notable peculiarity is that they among the few organisms which require an alkaline reaction as e of the principal conditions for their propagation.

So we see that it would be inadvisable to treat juices under these ditions with lime without an effective preventive, such as previous ting. By sulphitating the juice prior to liming, it is likely that propagation of the *Leuconostoc* germs is checked, and consequently the occurrence of dextran fermentation avoided.

Viscous Juices.—On the other hand, it is well known that one the peculiar characteristics of the "Uba" cane, which is the ble variety in Natal, is the production of extremely viscous juices, taining besides cane wax large quantities of gummy and pectin redients. Although under normal seasonal conditions the cane ces are comparatively pure, having a low percentage of glucose l being free of *Leuconostoc* germs, the excessive viscosity of the ce still remains.

The influence of these viscous substances upon the sugar solutions ater stages of the manufacture is of a no less damaging nature n those dealt with above, and it stands to reason that the early effective elimination of such ingredients is a matter of importance. e application of lime before sulphurous acid gas promotes a more rous precipitation of these viscous constituents, and would refore be more advisable on this account.

From this we see that in Natal, where the cane juices are of h a variable nature, either of the methods of sulphitation can be antageously applied according to the predominant condition.

Summary.—The results described in the above study, which are y confirmed by mill practice, show that there actually is a erence in the resulting product obtained from the two methods of phitation; they may not be observed in the laboratory, but this robably owing to the small quantities of chemicals used.

The Control of the Sulphitation of the Juice.

The control of the purification process of the crude juice, or in other words, the correct reaction of the juice after treatment with both agents, lime and sulphurous acid, is of the utmost importance, both in regard to effect on the subsequent stages of the manufacture, and the preservation of the installation and pipings.

Indicators.—Although the application of *litmus paper* as an indicator is undoubtedly the easiest and simplest method of testing the juice reaction, it must be borne in mind that apart from its unreliability in an atmosphere contaminated with acid gases, litmus reacts alkaline to acid sulphites, hence it is apt to make it difficult to determine accurately when the point of neutrality of the juice has been reached.

Phenolphthalein.—The only indicator of practical utility in sugar factories is phenolphthalein. This indicator may be applied in the form of air-dried paper or solution, the latter, owing to its superiority, being almost exclusively used. The phenolphthalein solution may be prepared according to different prescriptions, and consequently its composition varies in the different factories.

One method of preparing this indicator solution is to dissolve pure commercial phenolphthalein in a 90 per cent. alcohol solution, in the proportion of 1 to 30.

Another prescription,[1] which has the advantage of giving a more sensitive indicator, is as follows:

A 2 per cent. solution of phenolphthalein in a solution of alcohol (70 per cent.) is made, to which a few drops of dilute solution of potassium hydroxide are added. The bottle is then well shaken, to ensure thorough mixing. On the disappearance of the red colour, more drops of potassium hydroxide are added, until it is observed that the colour does not easily disappear, and that a faint coloration of the liquid may be noticed by holding it before a sheet of white paper. The solution is then ready to be used and should be preserved in closed bottles securely stoppered.

[1] Archief, 1908, p. 112

Testing for Neutrality.—The operation of testing the juice may described as follows :—[1]

"The detection of the very last, very slightly pink reaction is t always equally simple, and requires some routine. The best y of conducting this test is to place the juice and the indicator gether on a white porcelain plate, taking care that this does not ke place in an atmosphere charged with sulphurous acid, and ways in the same proportion, the decoloration of the solution in ese extremely sensitive tests being to a certain extent dependent on the quantity of the indicator solution employed . . ."

"It is, therefore, desirable always to bring together the same mber of drops of the juice and the same number of drops of the licator solution; for instance, three drops of juice and one drop of enolphthalein . . . Other methods favoured in factories consist placing a small quantity of the juice at the bottom of a porcelain sh when a few drops of neutral phenolphthalein solution are ded; or in placing a single drop of juice by means of a glass rod paper freshly moistened with the solution."

[1] "Plantation White Sugar Manufacture," Harloff & Schmidt, p 86

CHAPTER VIII.

SULPHITATION OF THE SYRUP AND OF MOLASSES.

The clear olive-yellow juice, on undergoing the concentration process in the evaporating installation, is converted into a turbid dark-coloured syrup. This change of colour invariably occurs during the concentration, so that even the best treated and clearest juices are bound to assume a darker colour on leaving the evaporation plant.

In order to manufacture plantation white sugar, it is imperative that the colour of the solution in which the crystallization of the sugar takes place should be as light as possible, for obvious reasons.

Thus besides the usual mechanical treatment of the syrup, consisting of the removal of impurities (which owing to the degree of concentration have not sufficient water to keep them in solution, and which have to be removed either by filtering or settling processes), it is manifest that the syrup requires to be decolorized prior to entering the vacuum pans.

Decolorizing Agents. -For this purpose various chemicals are used, including sulphurous acid, sodium acid phosphate, sodium hydrosulphite, etc.

Generally the application of sulphurous acid is preferred, owing to its cheapness and its powerful bleaching property. It is also usual to employ, in addition to the sulphurous acid, one of the other mentioned chemicals.

The sulphurous acid causes decolorization in two ways, as we have already noted. Firstly decolorization occurs from the reducing and consequent bleaching action of the sulphurous acid, and secondly it arises from the acid reaction imparted to the syrup, a decolorization engendered not only by SO_2 but by any acid more powerful than the organic acids present in the juice.

Sulphitation Operation.—The sulphitation of the concentrated juice or syrup may be conducted according to the "intermittent" or the "continuous" method.

Apparatus for Intermittent Sulphitation.—For the first mode of eration similar vessels to those dealt with in the chapter regarding n-juice sulphitation may be suitably applied. The main advantage these vessels is that they allow of an effective control of the lphitation process. A simpler and more convenient method is doubtedly the continuous sulphitation process. It may be carried t in an ordinary cylindrical vessel provided with a mixing arrange- ent or better in the following apparatus.[1]

Continuous Sulphitation Apparatus.—"The continuous sul- uring process of concentrated juice is effected by means of two tuation tanks. In the first tank the filtered concentrated juice is ughly sulphured to just below the required point for the maximum eaching, while but little sulphurous acid is admitted to the second nk in order clearly and easily to observe the final reactions.

"The supply of sulphurous acid to the first tank may remain altered, while the supply stop-cock of the second is turned on or according to requirement. In both tanks of course the juice ould be admitted from below, and run off at about the same level. rom the second tank it simply runs to the syrup supply tanks."

The sulphitation process generally occurs after the mechanical eatment of the syrup, *viz.*, elimination and either settling or tration, as for obvious reasons it is advisable to allow the syrup stand with a strong sulphurous acid reaction longer than is ecessary, or to raise its temperature above 65° C.

The Control of the Sulphitation of Syrup.

The extent of sulphitation to which the concentrated juice is bjected varies considerably in different factories and countries.

In some factories the colour of the acidified syrup is taken as a asis to indicate the extent of the sulphitation. In this case a mple of decolorized syrup acts as guide to the man in charge of e station.

Other factories, again, adopt litmus paper as an indicator, and onduct the sulphitating operation until a faint acid reaction is tained.

[1] "Plantation White Sugar Manufacture," Harloff & Schmidt, p. 109.

A practice much favoured in the manufacture of white sugar is to treat the syrup with sulphurous acid gas to an acidity of 1 to 1·4 grms. of SO_2 (and sulphites) per litre.

Determination of Extent of Sulphitation. For the purpose of controlling the sulphitation of syrup, the ordinary iodometric determination of sulphurous acid and its compounds is generally adopted.

The iodometric titration is based upon the oxidation of sulphurous acid and its salts by free iodine to sulphuric acid and sulphates. As indicator for this titration, a solution of starch is used, which on combining with free iodine to give starch iodide, assumes a blue colour. Thus by dropping the iodine solution into the acidified syrup, to which a little starch solution has previously been added, the blue coloration which is formed will disappear, at first quickly, but towards the end more slowly, until all the sulphurous acid and sulphites are converted, when further free iodine will form starch iodide, which causes the blue colour to be of a more permanent character.

Standard Iodine Solution.— With a view to simplicity in reading the degree of acidity, the iodine solution may be prepared as follows :-

Assume the acidity to which the syrup is to be sulphitated to be 1 grm. of SO_2 per litre. In using 10 c.c. of syrup for the titration it follows that these 10 c.c. correspond to 10 mgrms. of SO_2.

Further, according to the proportion :

127 iodine are equivalent to 32 SO_2

or (approximately) 4 iodine „ 1 SO_2

we find that in preparing a solution of 4 mgrms. of iodine to the c.c., one c.c. of the iodine solution used for the titration indicates 1 mgrm. of SO_2. Hence to test the sulphitation of 10 c.c. of syrup to an acidity of 1 grm. of SO_2 per litre (or 10 mgrm. of SO_2 for the 10 c.c. of syrup), 10 c.c. of the iodine solution are required, and similarly, for an acidity of 1·4 grms. of SO_2 per litre, 14 c.c. of the solution. We thus see that the number of cubic centimetres of the iodine solution used is ten times the number of grms. of SO_2 per litre of syrup. It is obvious that this arrangement considerably facilitates the reading of the titration.

The solution itself is prepared by dissolving 4 grms. of iodine in lution of about 7 grms. of potassium iodide dissolved in water, making the liquid up to 1000 c.c.

Iodine Titration Apparatus.—Taking into consideration the fact this process of titration in tropical cane sugar factories is usually

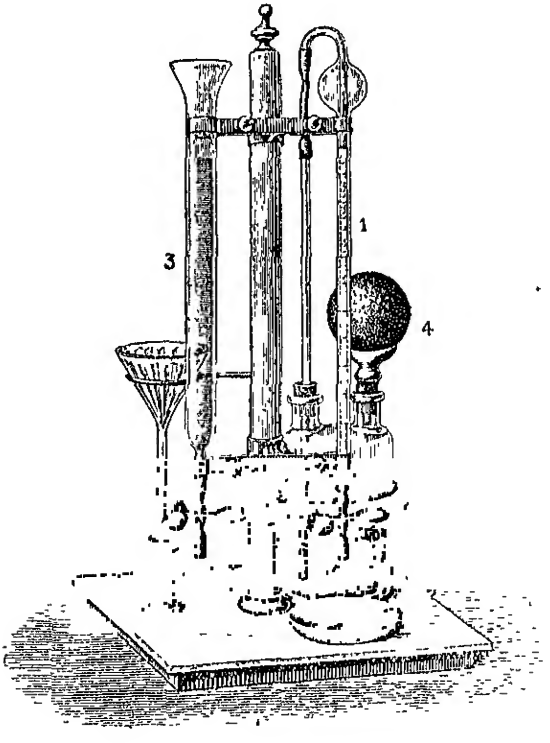

Fig. 6

Iodine Titration Apparatus

usted to an Asiatic man-in-charge, and further that it is of imance that the operation should be conducted rapidly, it is evident a practical and simple apparatus for this purpose should be pted.

A description of such an apparatus (Fig. 6) is as follows :—

The principal parts of the arrangement consist of a burette 1, a Wulff's bottle 2 containing the iodine solution, and a wider burette 3 filled with the starch solution. The connection between the first burette and the bottle is made by a syphon arrangement. A rubber cork carries a bent piece of glass tubing, the longer end of which reaches the bottom of the bottle, while the other end is melted into the wall of the burette, in such a manner that its mouth is on the same level as the zero mark.

By pressing the rubber ball 4 the iodine solution is forced through the glass tube into the burette, which is filled to a point a little above the zero mark. On releasing the rubber ball, the excess of the solution syphons back into the reservoir, leaving the burette filled exactly to the zero mark. The burette, which is generally graduated to one-tenth of a c.c., has a capacity to suit the circumstances. The regulation of the discharge of the titration liquid is effected either by a glass cock fixed on the burette or by a pinch cock acting on a rubber tube connecting the lower end of the burette with a pointed glass mouthpiece.

Method of Titration.—The process of titration may be carried out in an ordinary test tube, which is provided with a mark indicating a capacity of 10 c.c. The tube is filled to the mark with the syrup to be tested, to which a small quantity of the starch solution is added. Subsequently, the iodine solution in the burette is allowed to flow in small quantities into the test tube. This operation is repeatedly interrupted in order to thoroughly agitate the liquid in the tube. At first the blue coloration which is formed disappears immediately, but becomes gradually more permanent. The operation is repeated until the disappearance of the blue colour is effected only with difficulty on shaking the mixture, care being taken to add the iodine solution little by little as this point is approached and then only a drop at a time until the blue colour is finally fixed. The number of cubic centimetres of the solution required for the titration is read off the burette, indicating the degree of acidity according to the scheme previously outlined.

Simple Titration.—Another method of titration, which is to be
mmended owing to its simplicity, is the following:—

As a reservoir of the iodine solution, a simple bottle provided
a syphon, Fig. 7, is used. The titration is conducted in a

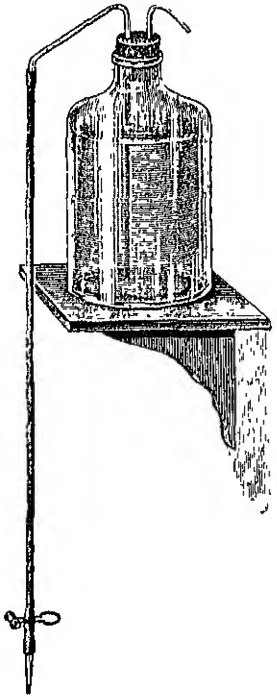

Fig. 7
Simple Titration Apparatus

Fig. 8
Vivien Tube

ien tube, (Fig. 8). After filling the tube to the zero mark
c.c.) with syrup, the same method of titration is carried out
e reading in this case is obtained from the graduated tube

Although the iodometric method is naturally not quite exact,
ng to the syrup containing other substances which will also be
dized by the iodine, it is sufficiently accurate and satisfactory for
l practice.

Sulphitation of Molasses.

Whether the sulphitation of the first molasses is justifiable or not is a matter of opinion. Some factories apply this process, others again find it more profitable to have recourse to other bleaching agents, such as sodium phosphate or sodium hydrosulphite.

The question of the sulphitation of molasses should be decided in each particular case. This may be done by laboratory experiments, namely by sulphitating a sample of molasses and examining the result.

In case this decolorization process is adopted, it is imperative that the molasses should be diluted prior to the operation, to approximately the concentration of the syrup. Mechanical treatment of the molasses, as in the case of the syrup, should precede the sulphitation process. The control of the operation is conducted in exactly the same way as with the syrup.

Inversion in Syrup.

Sulphurous Acid and Inversion.—It is a well-known fact that at a high temperature the action of sulphurous acid gas on pure sugar solutions is of an inverting character. However, it is equally established that in the case of cane (or beet) juices, which contain a certain quantity of organic salts, this inverting influence of the SO_2 is exerted to a much smaller extent. Thus the organic salts present in the syrup have a beneficial influence in checking the process of inversion. This property is attributed to the fact that these organic salts combine with the sulphurous acid and sulphuric acid occurring in the syrup, forming sulphites and sulphates, and liberating organic acids. Hence, the free mineral acids previously existing in the syrup are replaced by weak organic acids, the inverting power of which is considerably less.

Not only is the inversion process dependent upon the temperature, but also on the conditions under which the heating operation is performed. For instance, there is an appreciable difference between the results obtained from heating the sulphitated syrup at say 70° C, "in air" and "in vacuo."

n the first case the acidity of the syrup scarcely diminishes, the sulphurous acid (obviously occurring in the form of SO_3) is apt to be oxidized by the oxygen of the air forming huric acid. In the second case, however, this process is not y to occur, for these reasons :

. The acid reaction of the syrup in the vacuum pan diminishes lly with the volatilization of the free sulphurous acid and the nic acids.

2. The quantity of oxygen occurring in the pan is insufficient he above-mentioned process of oxidation.

We thus conclude that, provided the treatment of the syrup in sulphitation station is rationally conducted, no fear of extensive rsion need be entertained, though the syrup is strongly acid.

Conditions to be observed.—For the rational treatment of the p, the following points should be borne in mind.

1. The sulphitation operation should be carried out after the hanical treatment (filtering or settling) of the syrup.

2. The syrup to be sulphured should not be at a temperature ve 65° C, and if previous filtration is applied the syrup should ooled down to this temperature.

3. The time between the end of the sulphitation process and drawing of the acid syrup into the vacuum pans should be as t as possible.

4. All pipings through which acid syrup is to run should be le preferably of copper, and all reception tanks for acidified ip should be provided with a coating of acid-proof paint.

The last point, of course, has nothing to do with inversion, but t preventive measure against the contamination of the syrup n iron compounds, which impart a greyish colour to the sugar stals.

Corrosion in Syrup Apparatus.

The detrimental effect of corrosion, which invariably occurs to a ater or less extent when strongly acid syrups are dealt with in the uum pans, is familiar to those concerned with the manufacture vhite sugar.

During the boiling process, volatile organic acids and free sulphurous acid are carried along with the vapours, so that eventually the piping conducting these vapours is bound to suffer from the corrosive action of these acids.

In order to reduce the effect of corrosion to a minimum, it is advisable to coat the inside of the vapour-conducting piping and domes of the pans with a layer of ferric oxide.

Sodium Hydrosulphite, or "Blankit."

Its Use in Syrup Bleaching.—Since this subject belongs to the category of salts of sulphur acids, the treatment of this compound is within the scope of this work.

Blankit is the pure sodium salt of hydrosulphurous acid ($Na_2S_2O_4$) and comes into the market as a white powder, readily soluble in water; if protected from moisture, it will keep for an unlimited time in any climate.

The chemical action of sodium hydrosulphite on sugar juices is explained in two ways.[1]

According to the first equation, sulphurous acid gas and hydrogen are formed thus:

1. $Na_2S_2O_4 + H_2O = Na_2SO_4 + SO_2 + H_2$

and according to the second, under the influence of temperature, the hypothetical SO is formed, which is immediately oxidized to SO_2, thus.

2. $Na_2S_2O_4 = Na_2SO_4 + SO$
   $SO + H_2O = SO_2 + H_2$

The decolorization of the syrup by sodium hydrosulphite is instantaneous, and in spite of the small quantity of the agent used, the effect is striking.

Sodium hydrosulphite does not require an acid medium, but bleaches as well in neutral or alkaline solutions. The decolorization brought about by Blankit, however, is unstable, the original colour of the decolorized juices returning on exposure to the air.

[1] Deutsche Zuckerindustrie, 1908, p 735

this reason the agent should be applied at as late a stage he manufacture as possible, *viz.*, in the vacuum pan, shortly re graining.

A further advantage attributed to Blankit is that it diminishes viscosity of the syrup or molasses.

Application.—Sodium hydrosulphite may be applied either as a der or in solution. The latter is obviously to be preferred, as nds to a thorough mixing. It must be remembered, however, solutions of sodium hydrosulphite cannot be kept for long, as lose in decolorizing power through oxidation by the air, and t therefore always be made up fresh immediately before use.

In practice the application of Blankit is made in various ways. method is to add the Blankit to the concentrated juice not long ore it is boiled. Another, which is a most rational one, is to draw lution of Blankit into the vacuum pan before graining, and at ain intervals during the growth of the sugar crystals.

Syrup or Molasses—Order of Application of the different Agents.

Whether the Blankit should be applied to syrup or molasses, oth, is an open question, and greatly depends upon individual nion. While in Mauritius the author conducted a series of eriments touching this point.

Notable characteristics of Mauritian sugar are its brilliancy and teness. These qualities are indispensable if the sugar is to mpete with the Natal sugar on the Cape market. Now, as the hancy of the sugar is a prime factor, the investigation of this nt was one of the chief aims of the experiments

For this purpose syrups were separately treated with the three olorizing agents, *i.e.*, sulphurous acid, sodium phosphate and ium hydrosulphite, prior to entering the vacuum pans. The ars cured from the different strikes were examined as to whiteness brilliancy. The sugar originating from the syrup treated with ium hydrosulphite was by far the whitest, but at the same time

the least brilliant. Its appearance was comparable with that of snow, exceedingly white but dull. The syrup treated with sodium acid phosphate produced the most brilliant sugar, of a satisfactory white colour. In both cases the syrup was first sulphitated.

In the case of first molasses, on the other hand, the crystals are so small that there is no question of brilliancy, so that the whiteness of the sugar crystals became the prime factor. The very same experiments were carried out with the first molasses, the superiority of sodium hydrosulphite in this case being indisputable.

Thus we conclude that for the production of "superior plantation white sugar" it is advisable to treat the syrups with sulphurous acid and subsequently with sodium phosphate and the first molasses with sodium hydrosulphite.

CHAPTER IX.

THE SULPHITATION PROCESS IN PRACTICE.

Having studied the principle of the sulphitation process as ılied to cane juice, syrup and molasses, we can now proceed to vey the process as applied in practice, and, in the next chapter, iew "white sugar processes" in general.

General Scheme of Operation.—The usual modus operandi ɔpted by numerous white sugar mills follows with slight varia ıs the course given below.

Purification of the Mill Juice.—The mill juice runs from the l through copper strainers into two reception tanks, used alter- ely, in which some of the insoluble impurities are deposited by vity. These tanks are provided with overflows, which allow the e, now freed from its larger and heavier impurities, such as sand mud originally adhering to the cane, to discharge into a third ɔption tank. It is then pumped into measuring vessels, whence ravitates into liming tanks. Frequently the measuring tanks also used for liming purposes.

Tempering.—The tempering or liming of the juice is performed ıdding a definite quantity of lime-milk to the juice, the amount l varying considerably according to circumstances. It ranges ı about 6 to 12 litres (in exceptional cases even higher) of lime : of 15° Beaume to 1000 litres of juice. The usual practice ɔ use 8 litres, equivalent to 8 × 198 grms. of CaO. As it is ntial to obtain a thorough mixture of the lime milk with the e, the mixing process is best carried out in U-shaped stirring s fitted with a double spiral, gear-driven. The juice is then into a reception tank from which it is pumped into sulphitation els.

Sulphitation.—Under continuous agitation, the juice is sulphitated until neutrality with phenolphthalein is reached. Previous heating of the limed juice to about 45°C (in no case beyond 50°C) will prove beneficial.

Heating.—The sulphitated juice on leaving the sulphitation tanks is pumped through a multi-circulation quick-speed heater, in which the temperature of the juice is raised under pressure to about 105° C. A usual practice is to discharge the juice into a correction heater (Aspmall pan) provided with a steam heating device. The juice is here "corrected" from the somewhat varying temperature prevailing in the first heaters.

Cleaning.—It then runs by an overflow into a series of juice settling tanks or subsiders, where it is allowed to settle. The settling process is followed minutely by taking samples at intervals and observing the progress of the clarification of the juice.

The clear juice is tapped off from cocks, or float cocks, at suitable levels, and runs into a clear-juice tank, whence it is pumped into the evaporation plant. It is advisable to filter the juice prior to its entering the reception tank through a revolving sieve of very fine bronze gauze.

The sediment or dirty juice is allowed to discharge into blow-up tanks provided with a perforated copper coil, where it is diluted and boiled by means of live steam.

After a thorough boiling, the diluted dirty juice is pumped into a second set of settling tanks, having about one-fifth the capacity of the first tanks, and subjected to a second thorough subsidation.

The clear juice from these second subsiders is discharged into the same clear juice reception tank, but the sediment goes into a second series of blow-up tanks where it is diluted and boiled, and afterwards filtered in either filter-presses or Taylor filters. The clear juice from these filters runs into the clear juice tank.

Evaporation.—"If measures have been taken for settling the p, it should be borne in mind that the concentrated juice must be allowed to become too thick . . . A proper settling is proportate to the difference which exists between the density of the entrated juice and that of the subsiding impurities. If this rence is considerable, in other words, if the juice is less concend, settling will proceed much more readily, and *vice versa*."

"In general the impurities subside very readily and rapidly at Bé, but above 27·5° Bé the process is less successful." [1]

Treatment of the Syrup.—The syrup is drawn from the last el of the evaporating installation and discharged into eliminating s and heated to boiling point. The scum formed on the ace is carefully skimmed off, and the syrup allowed to gravitate ugh special filters into cooling tanks, or discharged into a series rup subsiders. The sediment in the latter case is preferably ned to the mill, or one of the first reception tanks, slowly and arly, so that it may be distributed as much as possible over volumes of the raw juice.

At a temperature below 65° C, the syrup is subsequently sulphitated acidity of 1·0 grm. of SO_2 per litre of concentrated juice and ped up into the vacuum pan supply tanks. An addition of sodium osulphite may be made in the tanks or in the pans at option.

Treatment of the First Molasses.

The molasses obtained on curing the first massecuites, of a purity to 68 and a Brix of about 80°, contains all the impurities of the ecuite in a more concentrated state, in addition to traces of iron ounds originating from the tanks by their constant contact the acid syrups and molasses. A separate clarification of the sses is therefore to be recommended, so that all the suspended anical impurities may be removed before a new crystallization place. These impurities may otherwise impart a dark tint to

[1] "Plantation White Sugar Manufacture" Harloff & Schmidt, p 112

the crystals of the second massecuite, and so prevent the obtaining of a superior product. An efficient clarification of the molasses is effected by the following method.

Clarifying the Molasses.—The molasses obtained on curing the first massecuites is pumped into eliminators provided with perforated steam coils. After being diluted to about 65° Brix (35·5 Be), the molasses is thoroughly boiled. The skimming off of the scum formed on the surface is most important and should be continually repeated.

The further treatment of the molasses, as in the case of the syrup, may be conducted in two ways, by (1) filtering, or (2) settling,

1. *Filtering*—The molasses is allowed to flow through special filters into tanks to cool down to a temperature below 65° C., at which temperature it is sulphitated to an acidity of 1·2 to 1·5 grms. of SO_2 per litre.

2. *Settling*—The molasses is discharged into subsiding tanks, where a quantity of sodium phosphate is added. The determination of the quantity required is carried out in the laboratory, where samples of the same molasses are treated with different quantities of the phosphate.

After having undergone either of these treatments the molasses is drawn into the vacuum pans. The application of sodium hydrosulphite in the vacuum pan will undoubtedly prove beneficial. The quantity of Blankit obviously varies according to circumstances, but approximately 17 to 20 grms. of this substance to the ton of massecuite may be applied.

Treatment of Second Molasses.—The molasses obtained on curing the second massecuites is diluted and thoroughly boiled in blow-up tanks, fitted with perforated copper coils. Generally a further clarification of this molasses is not required.

Boiling.

The treatment of this subject in detail would naturally be outside the scope of this work, hence only a summary is given in the following section:—

In Java, two methods of manufacturing white sugar are in vogue. The first produces only superior first sugar and superior

asses sugar, while the second method consists in turning out
r superior white sugar and exhausted molasses.

By keeping the first product separate from the after-product, it
vident that the quality of sugar is bound to be superior. There-
, in white sugar producing countries like Mauritius and Natal,
ere the colour of the sugars is of utmost importance, the method
btaining the whole product in one "jet" cannot be advantageously
pted.

The Two-sugar Method.—"In obtaining sugar in two jets, all
thick-juice is entirely boiled to a primary massecuite in such a
ner that the "run-offs" from it shall have a purity of about 65°.
se run-off syrups are then worked up by slow boiling and lengthy
ing to a molasses of about 33° quotient of purity."

'When the initial purity is high, the sugar is separated in three
es, *viz.* (1) first sugar with a run-off of 70°; (2) superior molasses
, with a run-off of 55°; and (3) molasses sugar. This last sugar
oun white simultaneously with the superior molasses sugar, and
ed with it."

Single Sugar Process.—"In the method of making white sugar
molasses, the purity of the thick-juice is continually lowered
mixing-in the first run-off. Generally, it is best to work with a
ture having a purity of 78°-80°, when the resulting massecuite
machining gives a run-off of about 58-60 purity. As above,
run-off is worked in one jet to molasses, obtaining so-called
lasses sugar,' which however is not machined white as before,
is pugged and drawn into the first sugar pans. The pugged
is diluted in the pans with some mixed thick juice, and
forms a 'pied-de-cuite' for starting grain for the first sugar
secuite."

'Such a first massecuite sugar consists of molasses sugar as
rimary grain, and is boiled with a mixture of syrup and thick-
having a purity of about 78°-80°. The remaining run-off once
n gives molasses sugar, etc."[1]

[1] antation White Sugar Manufacture" Harloff & Schmidt, pp. 128 and 129

For the manufacture of superior white sugar it is rational to adopt fractional centrifugalling, by which process the "green molasses" (impure molasses from the first set of centrifugals) is separated from the "clear molasses" (which is an almost pure sugar solution coming from the second set of centrifugals). The clear molasses is pumped either directly into the concentrated juice tanks or into special tanks, whence it is drawn into the pans separately. The green molasses is treated as explained previously.

Blueing the Sugars.

In order to neutralize the last traces of yellow tint on the crystals, it is a usual practice to blue the superior sugars with a solution of either ultramarine or indanthrene in the centrifugals. When employing this method, it is essential that the solution shall not contain large particles of the blueing substance in suspension.

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CHAPTER X.

ROCESSES ADOPTED BY THE LEADING WHITE SUGAR COUNTRIES.

After studying a general scheme of white sugar manufacture, can now proceed to deal with the main processes as applied in e three leading white sugar countries, *viz.*, Java, Mauritius and ıtal.

I. Java.

The scheme described in the previous chapter may be taken as e standard method of white sugar manufacture according to the phitation process in Java.

The Bach Treatment of Syrup.—Recently, however, a new ocess of treating the concentrated juice has been introduced by ch, and has given satisfactory results. The mode of operation ıy be described as follows :—

The syrup is drawn from the last vessel of the evaporation plant, d pumped through a syrup-cooler. The cooled syrup is discharged o a series of first sulphitation vessels, where it is first treated with ıe until a distinct alkaline reaction is reached, and the limed syrup ıbsequently sulphitated to neutrality. It then runs into eliminating ns, where it is heated to boiling point, the scum formed on the rface of the liquid being carefully skimmed off, and eventually e concentrated juice gravitates into a reception tank, whence it is mped through filter-presses.

The filtered syrup is pumped from a reception tank through a oler into a second set of sulphitation vessels. After being satis-ctorily bleached by the second application of the sulphurous acid s, the syrup is discharged into a reception tank and ultimately mped into the vacuum pan supply tanks, whence it is drawn into e pans.

II. Mauritius.

The general method of manufacturing white sugar in Mauritius is along the following lines:

Sulphitation.—The mill juice is pumped continuously though a sulphitation tank of the "continuous sulphitation" type, the sulphurous acid gas meanwhile being forced through the juice by means of steam injection (Giffard). The acidity to which the raw juice is generally sulphitated ranges from 0·8 to 1 per cent. SO_2.

Liming and Measuring.—The sulphitated juice is then run into liming tanks, in which it is measured and limed. The extent of liming varies in different factories: usually the acid juice is limed until only a slight acid reaction with litmus paper is obtained. Neutral juices are met with only in a few factories, the reason being the difficulty of obtaining an exact neutrality by means of lime. A few other factories, again, prefer to work with slightly acid juices. In some instances the sulphitated juice is rendered alkaline by a surplus of lime and subsequently treated with phosphoric acid until a slight acid reaction is reached.

Heating the Juice. The juice is pumped through a series of heaters, being usually heated to 70° 75° C. In a number of factories the juice, after being heated, is allowed to run through a "bac portal" (described in Chapter VII), in which it deposits part of its impurities. This apparatus is gradually becoming, however, less popular, and a great number of factories have discarded it.

Defecation.—From the "bac portal" the juice either runs or is pumped into defecators, provided with heating coils or double bottoms. It is heated to boiling point, in other words until the layer of scum formed on the surface begins to burst (cracking point), when the juice is allowed to settle.

Filtering.—The clear juice gravitates through a series of Danek filters, or in some cases through a sieve of fine gauze into a reception tank.

The sediment of the defecators is discharged into blow-up tanks, ıere it is thoroughly boiled, and afterwards pumped through filter-ɔsses, from which the filtered juice runs into the same reception ık.

Evaporation and Treatment with Phosphate.—This clear juice pumped into the evaporation plant and is concentrated to about '–28° Beaumé. The syrup is discharged into settling tanks of ıple capacity, where it is treated with phosphoric acid (in the 'm of calcium superphosphate containing 43–45 per cent. of osphoric acid). The amount of phosphate applied varies, ranging ɔm 1 to 1·5 grms. (in some cases even 2 grms.) of superphosphate ı litre of syrup.

Subsiding.—As this agent not only decolorizes the syrup, but ɔo produces an appreciable precipitate, it is evident that sufficient ne should be allowed for settling; according to the settling capacity the factory's disposal, the period ranges from 10 to 24 hours. The gree of concentration and the temperature of the syrup are of course ctors to be taken into consideration in deciding the period of the ttling process.

Sometimes the syrup from the evaporating installation is further ɔated to boiling point and "eliminated" prior to undergoing the ttling process. After the settling is completed, the sediment in the 'up subsiders is usually discharged into the juice defecators.

Boiling.—The clear concentrated juice is then drawn into the ıcuum pans. Often sodium hydrosulphite is applied to the syrup ther just before entering the pans or during the boiling.

As pointed out previously, the quality of the sugar in Mauritius mainly estimated on the colour of the product. Under these rcumstances it is not surprising that the process of "drawing in molasses in head boilings" to recover as much superior white ıgar as possible, is not favoured there.

The method generally adopted on that island is to produce "first ıgars" from syrup and "second sugars" from first molasses (both a superior white colour) without utilizing the "drawing in" ıocess. Further low grade sugars of an inferior colour are also roduced.

Molasses.—The molasses is treated in a manner previously described. It is thoroughly boiled in blow-up tanks and the lighter impurities carefully skimmed off. A certain quantity of phosphoric acid is added, and the molasses allowed to settle for a considerable time, the length of which depends upon circumstances.

Sodium hydrosulphite is also frequently applied in the pans, when boiling first molasses.

The crystallization and curing processes in the larger factories are usually carried out according to the methods now universally adopted.

III. Natal.

The manufacture of white sugar in this country is almost on the same lines as that in Mauritius. The following main differences, however, may be noted :-

Wax Separation.—Owing to the fact that the Natal juices (from "Uba" cane) generally contain excessive quantities of wax and other viscous substances, the limed and sulphitated juice is pumped into a series of defecators, better called "wax separators."

The majority of factories use the ordinary type of French defecators for this purpose, and follow the usual defecation process.

A more rational mode of operation now employed in several factories consists of allowing the juice to run through "continuous wax separators" composed of three or four defecators connected to one another. The juice runs slowly through the consecutive defecators, while the waxy matter rises to the surface to form a thick layer of scum, which is removed at frequent intervals. The temperature of the juice is kept below 82° C, as the wax melts at about that temperature, and the regulation of the temperature requires careful attention.

The clear juice running out from the last vessel is pumped through quick-speed heaters and its temperature raised to boiling point, when it gravitates into subsiding tanks and subsequently undergoes the same process as applied in Mauritius.

SUMMARY.

In conclusion, it may be useful to give a summary of the observations and conclusions arrived at in our study of the sulphitation process and the manufacture of "superior white sugar" according to that process. For convenience and facility of reference this can be done in the form of a series of questions and answers.

Tempering.

. In what form is the lime added to the juice?

1. As powdered quicklime, or a lime-milk.

. Which form is preferable, and for what reasons?

2. Lime-milk, as it conduces to thorough mixing. Unslaked lime is slaked incompletely in cold juice and is apt to form hard nodules covered with a sticky layer of impurities, which subsequently affect the quality of the juice.

. What quantity of lime-milk is used?

3. Usually 8 litres of lime-milk of 15° Beaumé per 1000 litres of juice. This figure varies considerably, and may range from 6 to 12 litres.

. How is the quantity of lime-milk determined?

4. (*a*) In the laboratory by determining how many c.cs. of lime-milk are necessary to obtain a complete precipitation in one litre of juice.

(*b*) The following method is preferable: Add a certain quantity of lime-milk to the mill juice and treat the latter

	in the factory according to the usual practice. A sample of the defecated juice is examined in the laboratory with a few drops of a solution of calcium saccharate. The formation of a precipitate indicates insufficient application of lime to the juice. The amount of lime-milk added to the mill juice is gradually increased until no further precipitate is obtained in the laboratory test. The absence of a precipitate in the first experiment shows that either the exact quantity or an excess of lime-milk has been applied: the regulation of the tempering should be performed accordingly.
5. Where and how is the juice to be treated with lime?	5. Preferably in special liming tanks under constant agitation. Another practice is to temper the juice in the measuring tank, whence it is discharged into limed juice stirring tanks
6. What form of agitating tank is most suitable?	6. Horizontal U-shaped tanks, provided with a double spiral, mechanically driven. Vertical tanks have been proved to allow the settling of lime at the bottom.
7. At what temperature should the tempering process be carried out?	7. The temperature should preferably be kept low, say about 30° 35° C.

Is it beneficial to heat the limed juice prior to sulphitation?

8. The heating of the limed juice immediately before sulphitation has given satisfactory results. The rise of temperature increases the energy of the liming action from the chemical point of view, so that gummy and pectin matters will be more readily precipitated. This is best done in the sulphitation vessels, which should be provided with heating coils. The resulting temperature should not exceed 50° C.

What is the action of lime on the juice?

9. It neutralizes the free acids occurring in the juice, acts on albuminoid, gummy and pectin substances, which are subsequently partly removed, precipitates various bark pigments and chromogens and forms saccharates and glucosates.

Preparation of Lime-milk.

10. What are the qualities of the lime to be used?

10. The lime should be of the purest and best kind available, containing as little impurity as possible. The maximum of magnesia in the lime allowable is 2 per cent. If over burned, the lime becomes rocky and slakes with difficulty, so that it loses its efficiency. The lime should not contain any unburned particles, as these cause trouble.

11. Of what nature should the lime-milk be?

11. The lime-milk should be homogeneous and void of hard particles of lime, which are apt to escape the action of the sulphurous acid and afterwards gradually dissolve, imparting an alkaline reaction to the juice.

It is therefore essential that the lime should be thoroughly powdered and slaked.

Sulphitation of the Juice.

12. What form of sulphitation vessel is to be preferred?

12. Vertical cylindrical vessels, provided with heating coils, agitating device, gas distributing piping and further accessories, such as gauge glasses, test cocks, thermometers, etc.

13. How is the distribution of the saturation gas performed?

13. Either by means of perforated pipes or by so-called "spider webs." The efficiency of the SO_2 gas is obviously dependent upon the thoroughness of its distribution and also upon the height of the juice to be treated. Hence these points should be taken into consideration when designing a sulphitation vessel.

14. How can the mixing process of the juice and the gas be assisted?

14. By continuous agitation of the juice during the sulphitating operation and also by allowing the juice and gas to enter into the tank through a common pipe.

How is the sulphitation process controlled?

15. By the use of indicators, usually litmus or phenolphthalein, in the form of sensitized paper or solution. The phenolphthalein solution is preferable.

A supplementary control of the sulphitation process may be advantageously carried out "by sight," that is, by following the course of sulphitation and taking samples at intervals, also observing the process of settling.

How is the phenolphthalein solution prepared?

16. By dissolving phenolphthalein in a 90 per cent. alcohol solution, in the proportion of 1 to 30.

How is the indicator solution applied?

17. By adding to a few drops of juice in a white porcelain dish one or two drops of the phenolphthalein solution, or by placing a single drop of the juice by means of a glass rod on paper freshly moistened with the solution.

To what extent is the sulphitation process carried out?

18. Until the indicator solution assumes only a faint pink coloration, instead of a deep crimson.

What occurs if the sulphitation is further extended?

19. "Over-sulphitation," which renders the juice acid and causes the formation of soluble bisulphites, which are subsequently decomposed in the evaporators into insoluble sulphites and free sulphurous acid.

20. To what is incrustation and corrosion attributed?	20. Chiefly to the above-mentioned decomposition. The sulphites form incrustations on the tubes of the calandrias, while the liberated sulphurous acid is converted into sulphuric acid which exerts its corrosive properties. Free sulphurous acid is also produced by volatilization.
21. How does inversion occur?	21. When acid juices are subjected to high temperatures inversion takes place.
22. For what purpose is the sulphurous acid gas used?	22. In the case of limed juices, SO_2 is chiefly used as neutralizer.

Generation of Sulphurous Acid.

23. In what form is the sulphurous acid used?	23. In cane sugar factories almost exclusively in the gaseous form.
24. How is it obtained?	24. By the simple combustion of sulphur in air; $S + O_2 \quad SO_2$
25. What is the most suitable installation for the production of SO_2?	25. The combination shown in Fig. 3, Chapter III, composed of air-drier, air-compressor, air-recipient, sulphur furnace, gas purifier and cooler.
26. What are its advantages over other plants?	26. Mainly the generation of very pure saturation gas, and an easy control of the operation.
27. What is the usual proportion of SO_2 in the combustion gas?	27. This figure varies considerably, but 15 per cent. is satisfactory.

8. What are the main points to be borne in mind in the generation of SO_2?

28.(*a*) Dry air for combustion.
(*b*) Air supply to be regular.
(*c*) Combustion temperature in the furnace to be kept as low as possible.
(*d*) Uniform combustion of the sulphur layer in the furnace.
(*e*) Sublimation in the pipings to be checked as much as possible.
(*f*) The temperature of the saturation gas to be kept low.
(*g*) The gas to be purified before entering the sulphitation vessels.

9. How are the precautionary measures to be carried out?

29.(*a*) By drawing the air through an air dryer, composed of several layers of quicklime.
(*b*) By using an air-recipient and regulating valves.
(*c*) By cooling the furnace by means of continuously running water.
(*d*) By melting the sulphur rolls in the furnace by previous heating of the tray, as explained in Chapter IV.
(*e*) By cooling, and the provision of sublimators.
(*f*) By constant cooling of the gas by means of water-jackets.
(*g*) By filtering the gas through coke or pumice stone, and washing to absorb free SO_3.

Treatment of the Syrup and Molasses.

30. How is the syrup treated prior to sulphitation?

30. The syrup is heated to boiling point, eliminated, and either filtered or settled. If filtering is employed, subsequent cooling of the syrup is necessary.

31. How is the sulphitation carried out?

31. The syrup is pumped up into sulphitation vessels and treated with sulphurous acid gas until an acidity of 1 to 1·2 grms. of SO_2 per litre is obtained.

32. How is the sulphitation of the syrup controlled?

32. By means of an iodine solution, with a starch solution as indicator.

33. How is a standard iodine solution prepared?

33. By dissolving 4 grms. of iodine and about 7 grms. of potassium iodide in 1 litre of water.

34. How is the titration carried out with the iodine solution?

34. 10 c.c. of syrup are diluted with water, a little of the starch solution added, and subsequently titrated with the iodine solution. Each c.c. of the standard iodine solution indicates 1·0 mgrm. SO_2.

35. What is the action of SO_2 on syrup?

35. The action of SO_2 on syrup is fourfold, namely:

(*a*) It reduces the organic colouring substances occurring in the syrup to colourless compounds. The bleaching effect due to this phenomenon is, however, only of a temporary nature, as by oxidation the original dark colour of the syrup gradually returns.

(*b*) It imparts an acid reaction to the syrup, by which a decolorization is caused. This decolorization is preserved so long as the syrup remains acid.

(*c*) It exercises a purifying influence, as it decomposes part of the non-sugars, which may be removed eventually.

(*d*) It further acts as a preservative of the syrup.

How does the question of inversion enter here?

36. Although the syrup has a distinctly acid reaction, a rational treatment of the syrup after sulphitation precludes inversion to any extent.

At what temperature is the sulphitation process to take place?

37. It is advisable not to allow the temperature of acid syrups to exceed 65° C in the atmosphere. A rise in the temperature in the vacuum pans is of no consequence, as already pointed out.

How is the molasses treated?

38. The first molasses is diluted to about the density of the syrup, and subsequently treated in the same way as the syrup. The application of additional bleaching agents, such as phosphates or sodium hydrosulphites may prove to be advantageous.

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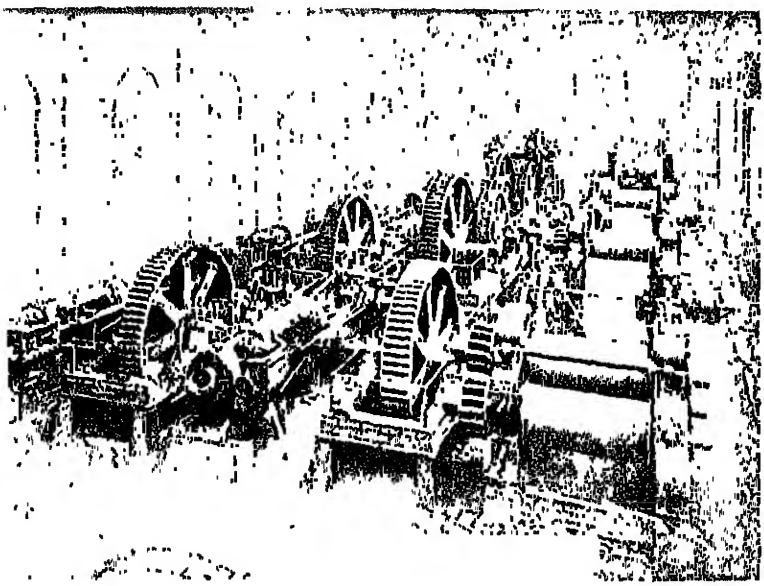

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